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1-(2-Chlorobenzoyl)-3-(pyrimidin-2-yl)-thiourea

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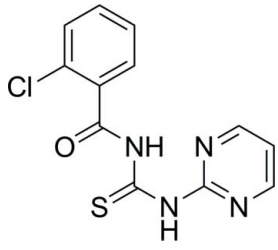
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.082; data-to-parameter ratio = 16.0.

In the title compound, $C_{12}H_9ClN_4OS$, the carbonyl group is at a *cis* position with respect to the thiourea unit. The dihedral angle between the phenyl and pyrimidine ring is 16.49 (6)°. An intramolecular N—H···N hydrogen bond stabilizes the molecular conformation. In the crystal, N—H···N, C—H···O and C—H···S hydrogen bonds generate chains along the *bc* axis.

Related literature

For background to our work on structural and coordination chemistry of *N*,*N*′-disubstituted thioureas, see: Rauf *et al.* (2012). For a related structure, see: Sultana *et al.* (2007).



Experimental

Crystal data

$C_{12}H_9ClN_4OS$
M_r = 292.74
Triclinic, $P\overline{1}$
a = 7.167 (3) Å
b = 8.000 (4) Å
c = 11.252 (5) Å
α = 81.625 (14)°
β = 74.580 (12)°
γ = 83.979 (15)°
V = 613.8 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 123 K
0.30 × 0.26 × 0.18 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
4888 measured reflections
2759 independent reflections
2590 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.082
S = 1.07
2759 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3	0.88	1.93	2.611 (2)	133
N2—H2···N4^{i}	0.88	2.21	3.068 (2)	166
C11—H11···O1^{ii}	0.95	2.28	3.200 (2)	163
C12—H12···S1^{i}	0.95	2.77	3.568 (2)	142

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $x-1, y+1, z$.

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2001); cell refinement: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2001); data reduction: *CrystalClear*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *Yadokari-XG 2009* (Kabuto *et al.*, 2009).

MKR is grateful to The Quaid-i-Azam University, Islamabad, for financial support of a postdoctoral fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2612).

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supporting information

Acta Cryst. (2013). E**69**, o84 [https://doi.org/10.1107/S1600536812050118]

1-(2-Chlorobenzoyl)-3-(pyrimidin-2-yl)thiourea

M. Khawar Rauf, Samad Yaseen, Masahiro Ebihara and Amin Badshah

S1. Comment

In continuation of our work on the structural and coordination chemistry of *N*,*N*′-disubstituted thioureas (Rauf *et al.*, 2012) the structure of the title compound (Fig. 1) is described in this article. The bond lengths and angles in the title compound agree very well with the corresponding bond legths and angles reported in a closely related compound (Sultana *et al.*, 2007). The molecule exists in its thione form with typical thiourea C—S and C—O bond distances, as well as shortened C—N bonds. The plane containing the S1, C2, N1 & N2 atoms is almost parallel to the pyrimidine ring, forming a dihedral angle of 9.09 (13)°. The molecules also feature intra & intermolecular N—H···N, C—H···O and C—H···S hydrogen bonds (Table 1 & Fig. 2).

S2. Experimental

Freshly prepared 2-chlorobenzoylisothiocyanate (1.98 g, 10 mmol) was dissolved in tetrahydrofuran (35 ml) and stirred for 40 minutes. Afterwards neat 2-aminopyrimidine (1.0 g, 10 mmol) was added and the resulting mixture was stirred for 1 h. The reaction mixture was then poured into acidified water and stirred well. The solid product was separated and washed with deionized water and purified by recrystallization from chloroform to give fine crystals of the title compound, with an overall yield of 92% (2.8 g).

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 and N—H = 0.88 Å, and U_{iso}(H) set at 1.2U_{eq}(C/N).

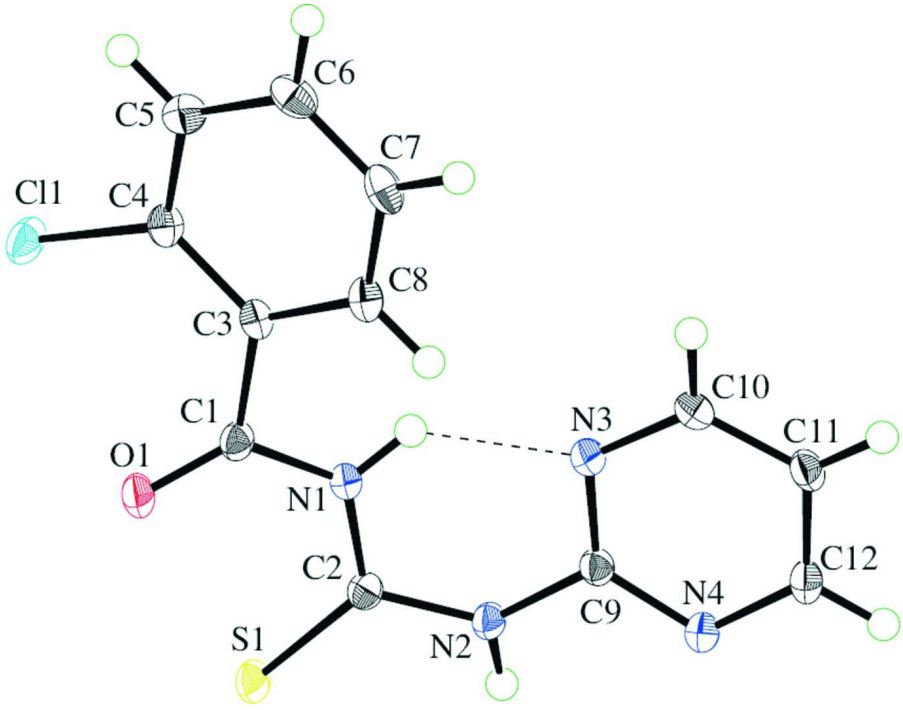

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius. Hydrogen bond is shown as a dashed line.

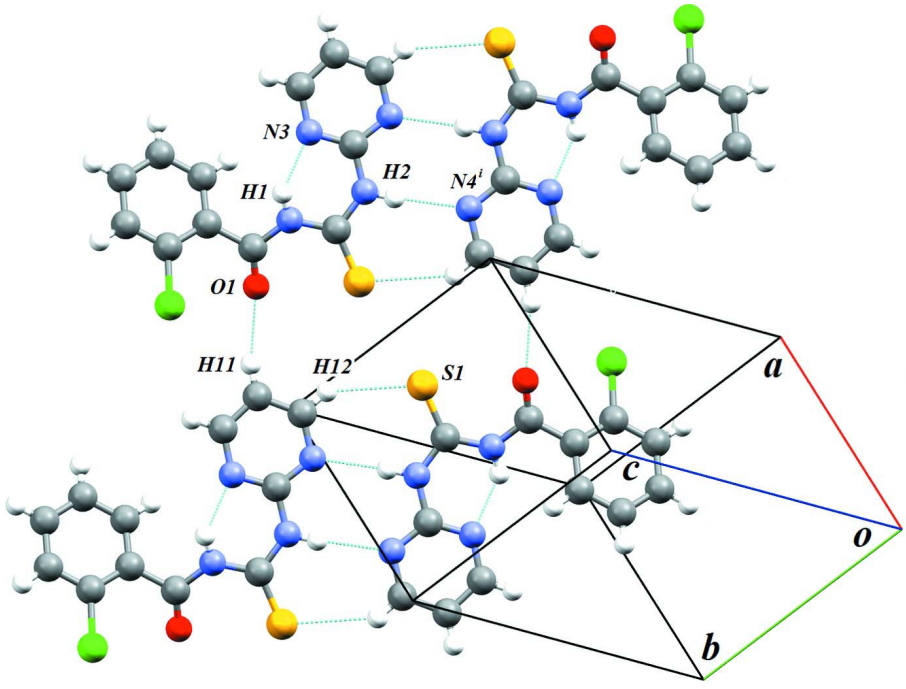

Figure 2

A view of the N—-H···N, C—H···O and C—H···S hydrogen bonds (dotted lines) in the crystal structure of the title compound.

1-(2-Chlorobenzoyl)-3-(pyrimidin-2-yl)thiourea

Crystal data

$C_{12}H_9ClN_4OS$
$M_r = 292.74$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.167$ (3) Å
$b = 8.000$ (4) Å
$c = 11.252$ (5) Å
$\alpha = 81.625$ (14)°
$\beta = 74.580$ (12)°
$\gamma = 83.979$ (15)°
$V = 613.8$ (5) Å^3
$Z = 2$
$F(000) = 300$
$D_x = 1.584$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 2102 reflections
$\theta = 3.0$–$27.5°$
$\mu = 0.48$ mm^{-1}
$T = 123$ K
Block, yellow
0.30 × 0.26 × 0.18 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
Radiation source: Rotating Anode
Graphite Monochromator monochromator
Detector resolution: 14.62 pixels mm^{-1}
ω scans
4888 measured reflections
2759 independent reflections
2590 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -8 \rightarrow 9$
$k = -9 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.082$
$S = 1.07$
2759 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + 0.4951P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.5699 (2)	0.40086 (19)	0.77222 (14)	0.0168 (3)
O1	0.66497 (17)	0.29064 (14)	0.81927 (11)	0.0232 (3)
N1	0.54980 (19)	0.57016 (16)	0.79213 (12)	0.0174 (3)
H1	0.4808	0.6385	0.7489	0.021*
C2	0.6242 (2)	0.64410 (19)	0.87083 (14)	0.0167 (3)

S1	0.80674 (6)	0.56064 (5)	0.93013 (4)	0.02194 (12)
N2	0.53983 (18)	0.80178 (16)	0.89581 (12)	0.0172 (3)
H2	0.5987	0.8564	0.9362	0.021*
C3	0.4553 (2)	0.36848 (18)	0.68461 (14)	0.0160 (3)
C4	0.5267 (2)	0.25791 (19)	0.59525 (15)	0.0175 (3)
C5	0.4187 (2)	0.2281 (2)	0.51587 (15)	0.0206 (3)
H5	0.4713	0.1549	0.4540	0.025*
C6	0.2331 (2)	0.3058 (2)	0.52718 (16)	0.0225 (3)
H6	0.1586	0.2857	0.4730	0.027*
C7	0.1569 (2)	0.4122 (2)	0.61713 (16)	0.0222 (3)
H7	0.0290	0.4634	0.6260	0.027*
C8	0.2671 (2)	0.44417 (19)	0.69453 (15)	0.0190 (3)
H8	0.2141	0.5187	0.7554	0.023*
Cl1	0.75833 (5)	0.15669 (5)	0.57492 (4)	0.02521 (12)
C9	0.3753 (2)	0.88839 (19)	0.86695 (14)	0.0162 (3)
N3	0.2989 (2)	0.83057 (17)	0.78621 (13)	0.0208 (3)
C10	0.1374 (2)	0.9160 (2)	0.76400 (15)	0.0215 (3)
H10	0.0770	0.8766	0.7087	0.026*
C11	0.0568 (2)	1.0582 (2)	0.81851 (15)	0.0191 (3)
H11	−0.0576	1.1179	0.8028	0.023*
C12	0.1518 (2)	1.10993 (19)	0.89792 (15)	0.0185 (3)
H12	0.1015	1.2096	0.9356	0.022*
N4	0.31077 (18)	1.02648 (16)	0.92412 (12)	0.0169 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0139 (7)	0.0176 (7)	0.0177 (7)	0.0017 (5)	−0.0021 (5)	−0.0037 (6)
O1	0.0235 (6)	0.0197 (5)	0.0287 (6)	0.0079 (4)	−0.0125 (5)	−0.0064 (5)
N1	0.0190 (6)	0.0162 (6)	0.0180 (6)	0.0043 (5)	−0.0074 (5)	−0.0039 (5)
C2	0.0149 (7)	0.0182 (7)	0.0160 (7)	0.0011 (5)	−0.0025 (5)	−0.0029 (5)
S1	0.01718 (19)	0.0234 (2)	0.0285 (2)	0.00729 (14)	−0.01126 (16)	−0.01008 (16)
N2	0.0159 (6)	0.0175 (6)	0.0198 (6)	0.0030 (5)	−0.0070 (5)	−0.0060 (5)
C3	0.0147 (7)	0.0139 (6)	0.0184 (7)	0.0000 (5)	−0.0043 (6)	0.0001 (5)
C4	0.0136 (7)	0.0165 (7)	0.0210 (7)	−0.0011 (5)	−0.0028 (6)	−0.0009 (6)
C5	0.0217 (8)	0.0207 (7)	0.0197 (7)	−0.0044 (6)	−0.0048 (6)	−0.0024 (6)
C6	0.0210 (8)	0.0240 (8)	0.0248 (8)	−0.0064 (6)	−0.0112 (6)	0.0029 (6)
C7	0.0154 (7)	0.0210 (7)	0.0298 (8)	−0.0003 (6)	−0.0085 (6)	0.0029 (6)
C8	0.0156 (7)	0.0166 (7)	0.0235 (8)	0.0009 (6)	−0.0041 (6)	−0.0010 (6)
Cl1	0.01698 (19)	0.0298 (2)	0.0311 (2)	0.00670 (15)	−0.00699 (15)	−0.01528 (17)
C9	0.0153 (7)	0.0164 (7)	0.0156 (7)	0.0008 (5)	−0.0029 (5)	−0.0011 (5)
N3	0.0226 (7)	0.0209 (6)	0.0213 (7)	0.0062 (5)	−0.0107 (5)	−0.0063 (5)
C10	0.0228 (8)	0.0230 (8)	0.0210 (8)	0.0032 (6)	−0.0113 (6)	−0.0035 (6)
C11	0.0160 (7)	0.0197 (7)	0.0202 (7)	0.0033 (6)	−0.0053 (6)	0.0001 (6)
C12	0.0165 (7)	0.0151 (7)	0.0212 (7)	0.0023 (5)	−0.0021 (6)	−0.0010 (6)
N4	0.0155 (6)	0.0157 (6)	0.0189 (6)	0.0005 (5)	−0.0036 (5)	−0.0026 (5)

Geometric parameters (Å, º)

C1—O1	1.2051 (19)	C6—C7	1.380 (2)
C1—N1	1.391 (2)	C6—H6	0.9500
C1—C3	1.504 (2)	C7—C8	1.386 (2)
N1—C2	1.374 (2)	C7—H7	0.9500
N1—H1	0.8800	C8—H8	0.9500
C2—N2	1.374 (2)	C9—N3	1.334 (2)
C2—S1	1.6596 (16)	C9—N4	1.338 (2)
N2—C9	1.3937 (19)	N3—C10	1.344 (2)
N2—H2	0.8800	C10—C11	1.371 (2)
C3—C4	1.395 (2)	C10—H10	0.9500
C3—C8	1.403 (2)	C11—C12	1.386 (2)
C4—C5	1.386 (2)	C11—H11	0.9500
C4—Cl1	1.7420 (17)	C12—N4	1.339 (2)
C5—C6	1.390 (2)	C12—H12	0.9500
C5—H5	0.9500		
O1—C1—N1	125.24 (14)	C7—C6—H6	120.0
O1—C1—C3	122.86 (14)	C5—C6—H6	120.0
N1—C1—C3	111.90 (12)	C6—C7—C8	119.97 (15)
C2—N1—C1	128.18 (13)	C6—C7—H7	120.0
C2—N1—H1	115.9	C8—C7—H7	120.0
C1—N1—H1	115.9	C7—C8—C3	121.20 (15)
N1—C2—N2	114.75 (13)	C7—C8—H8	119.4
N1—C2—S1	125.09 (12)	C3—C8—H8	119.4
N2—C2—S1	120.12 (12)	N3—C9—N4	126.22 (14)
C2—N2—C9	129.89 (13)	N3—C9—N2	118.96 (14)
C2—N2—H2	115.1	N4—C9—N2	114.82 (13)
C9—N2—H2	115.1	C9—N3—C10	116.63 (14)
C4—C3—C8	117.63 (14)	N3—C10—C11	122.19 (15)
C4—C3—C1	122.04 (13)	N3—C10—H10	118.9
C8—C3—C1	120.26 (14)	C11—C10—H10	118.9
C5—C4—C3	121.41 (14)	C10—C11—C12	116.25 (14)
C5—C4—Cl1	117.02 (12)	C10—C11—H11	121.9
C3—C4—Cl1	121.54 (12)	C12—C11—H11	121.9
C4—C5—C6	119.70 (15)	N4—C12—C11	123.30 (14)
C4—C5—H5	120.2	N4—C12—H12	118.3
C6—C5—H5	120.2	C11—C12—H12	118.3
C7—C6—C5	120.06 (15)	C9—N4—C12	115.35 (13)
O1—C1—N1—C2	−3.0 (3)	C4—C5—C6—C7	0.0 (2)
C3—C1—N1—C2	176.24 (14)	C5—C6—C7—C8	1.3 (2)
C1—N1—C2—N2	−163.48 (14)	C6—C7—C8—C3	−0.9 (2)
C1—N1—C2—S1	18.9 (2)	C4—C3—C8—C7	−0.9 (2)
N1—C2—N2—C9	9.3 (2)	C1—C3—C8—C7	−178.10 (14)
S1—C2—N2—C9	−172.89 (13)	C2—N2—C9—N3	−12.7 (2)
O1—C1—C3—C4	−39.8 (2)	C2—N2—C9—N4	168.18 (15)

N1—C1—C3—C4	140.94 (14)	N4—C9—N3—C10	−2.7 (2)
O1—C1—C3—C8	137.32 (16)	N2—C9—N3—C10	178.32 (14)
N1—C1—C3—C8	−41.99 (19)	C9—N3—C10—C11	1.7 (2)
C8—C3—C4—C5	2.2 (2)	N3—C10—C11—C12	0.2 (2)
C1—C3—C4—C5	179.40 (14)	C10—C11—C12—N4	−1.5 (2)
C8—C3—C4—Cl1	−179.94 (11)	N3—C9—N4—C12	1.5 (2)
C1—C3—C4—Cl1	−2.8 (2)	N2—C9—N4—C12	−179.46 (13)
C3—C4—C5—C6	−1.8 (2)	C11—C12—N4—C9	0.7 (2)
Cl1—C4—C5—C6	−179.74 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3	0.88	1.93	2.611 (2)	133
N2—H2···N4^{i}	0.88	2.21	3.068 (2)	166
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C12—H12···S1^{i}	0.95	2.77	3.568 (2)	142

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+2; (ii) *x*−1, *y*+1, *z*.